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Resolution of a modulated structure by electron and powder X-ray diffraction

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These past few years, many new structures have been solved using electron diffraction methods: zone axis precession electron diffraction (PED) and tomography in reciprocal space. Both methods enable to reduce importantly the multiple scattering, so that the reflection intensities can be used for structure determination by direct methods. The ferrite Sr25Fe30O77 belongs to a family of phases whose structures consist of an intergrowth of m perovskite layers with complex rocksalt type layers [1,2]. The compound of interest is the member m = 4 of this family and its structure has been solved by combining both electron diffraction methods cited above. This oxide crystallizes in an orthorhombic system with the sub-cell parameters a ≈ b ≈ 5.4 Å and c ≈ 42 Å in a F type lattice. The structure exhibits modulation along a axis with a modulation vector q = 2/5 a. The commensurate nature of the modulation enables to describe the structure in a supercell with the cell parameters a ≈ 27 Å, b ≈ 5.4 Å and c ≈ 42 Å. PED patterns were recorded in zone axis with a Spinning Star unit using a precession angle of 2°. The intensities were extracted with CRISP software and the resulting dataset was then implemented in SIR2008 for structure solution. The tomography data collection, recorded by tilting manually every 0.5 degree from -30 to +30 degrees, was inserted in a "3D Electron Diffraction Tomography" software, which reconstructs the 3D reciprocal space and extracts automatically the reflection intensities. The intensity file was then used on SIR2008 for structure determination. In order to confirm and refine the structural model, a powder X-ray diffraction pattern was performed on a laboratory diffractometer with Cu Ka1 radiation. Cell parameters were refined with the WinPlotr and FullProf Softwares using both LeBail and Rietveld methods [3]. The structural model obtained with electron diffraction data was tried and confirmed as the correct structure by the Rietveld refinements.

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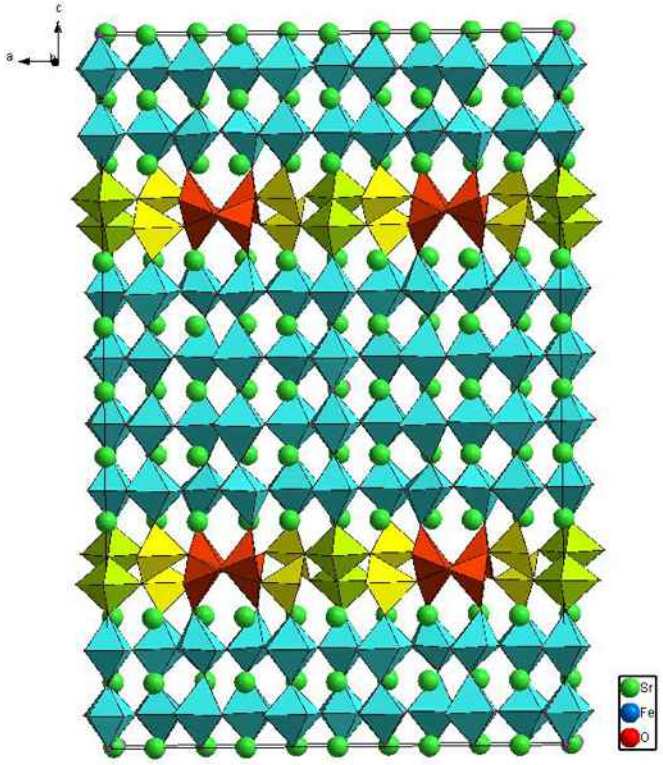

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